

**UNITED STATES AIR FORCE  
ARMSTRONG LABORATORY**

**PRELIMINARY ANALYSIS OF  
OFF-GASSING FOLLOWING THERMAL  
STRESS OF ADVANCED COMPOSITE  
MATERIALS**

**Gary D. Meyer  
John C. Lipscomb**

**OPERATIONAL TOXICOLOGY BRANCH  
HUMAN EFFECTIVENESS DIRECTORATE  
2856 G STREET, BLDG 79  
WRIGHT-PATTERSON AFB OH 45433-7400**

**David L. Courson**

**MANTECH - GEO-CENTERS JOINT VENTURE  
P.O. BOX 31009  
DAYTON, OH 45437-0009**

**August 1998**

**Air Force Research Laboratory  
Human Effectiveness Directorate  
Crew Survivability and Logistics Division  
Operational Toxicology Branch  
2856 G Street  
Wright-Patterson AFB OH 45433-7400**

Approved for public release; distribution is unlimited.

**DTIC QUALITY INSPECTED 4**

**19990902 066**

## NOTICES

When US Government drawings, specifications or other data are used for any purpose other than a definitely related Government procurement operation, the Government thereby incurs no responsibility nor any obligation whatsoever, and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise, as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Please do not request copies of this report from the Air Force Research Laboratory. Additional copies may be purchased from:

National Technical Information Service  
5285 Port Royal Road  
Springfield, Virginia 22161

Federal Government agencies and their contractors registered with the Defense Technical Information Center should direct requests for copies of this report to:

Defense Technical Information Service  
8725 John J. Kingman Rd., Ste 0944  
Ft. Belvoir, Virginia 22060-6218

### DISCLAIMER

This Technical Report is published as received and has not been edited by the Technical Editing Staff of the Air Force Research Laboratory.

### TECHNICAL REVIEW AND APPROVAL

**AFRL-HE-WP-TR-1998-0083**

This report has been reviewed by the Office of Public Affairs (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

### FOR THE DIRECTOR



**STEPHEN R. CHANNEL**, Maj, USAF, BSC  
Branch Chief, Operational Toxicology Branch  
Air Force Research Laboratory

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE August 1998		3. REPORT TYPE AND DATES COVERED Interim Report
4. TITLE AND SUBTITLE Preliminary Analysis of Off-Gassing Following Thermal Stress of Advanced Composite Materials			5. FUNDING NUMBERS Contract No: F41624-96-C-9010 PE 62202F PR 7757 TA 7757A2 WU 7757A103	
6. AUTHOR(S) Meyer, G.D.; Lipscomb, J.C.; Courson, D.L.				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) ManTech - GEO-CENTERS Joint Venture Toxic Hazards Research P.O. Box 31009 Dayton, OH 45437-0009			8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Air Force Research Laboratory, Human Effectiveness Directorate Crew Survivability and Logistics Division, Operational Toxicology Branch AFRL/HEST Bldg 79 2856 G Street Wright-Patterson AFB, OH 45433-7400			10. SPONSORING/MONITORING AGENCY REPORT NUMBER  AFRL-HE-WP-TR-1998-0083	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION AVAILABILITY STATEMENT  Approved for public release; distribution is unlimited			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Advanced composite material (ACM) use in the military and private sectors is increasing. Previous work identified the release of toxic compounds during combustion of ACM. However, the question of off-gassing following heat stress had not been addressed. This preliminary study looked at the semivolatile compounds off-gassed from four different types of ACM following thermal stress of 625°C. Daily samples of the off-gassed semivolatiles were collected by Tenax TAØ tubes and by cold trapping. The ACM samples were still off-gassing at the end of the 15-16 day sampling period. Results with the four targeted compounds (aniline, phenol, quinoline, and naphthalene) and the semivolatile compounds tentatively identified by ion match were too varied to prudently extrapolate the identity and/or quantity released of any specific compounds associated with another type of ACM. However, it can be deduced that only well-ventilated areas should be used for thermally stressed ACM to prevent the build-up of off-gassed materials to dangerous levels. In addition, the study results fully support the continued use of the personal protective equipment guidelines presented in paragraph 3-4,e,(3) of TO 00-105E-9, Aircraft Emergency Response. These results underscore the need to examine the specific ACM in question and address the issues associated with that ACM. Further research is recommended to fully characterize ACM off-gassing following thermal stress.				
14. SUBJECT TERMS Combustion      Off-Gassing      Aniline      Phenol Quinoline      Naphthalene      Advanced Composite Material (ACM)			15. NUMBER OF PAGES	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT  UNCLASSIFIED		18. SECURITY CLASSIFICATION OF THIS PAGE  UNCLASSIFIED		19. SECURITY CLASSIFICATION OF ABSTRACT  UNCLASSIFIED
				20. LIMITATION OF ABSTRACT  UL

**THIS PAGE INTENTIONALLY LEFT BLANK.**

## TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
List of Figures and Tables .....	iv
Preface .....	v
Abbreviations .....	vi
Introduction .....	1
Materials and Methods .....	2
Results .....	4
Summary .....	5
References .....	6
Appendix .....	7

## LIST OF FIGURES AND TABLES

<u>Title</u>	<u>PAGE</u>
Figure A-1. Canister .....	7
Figure A-2. Cold Trap Set-Up .....	8
Table 1. Average reduction of sample ACM mass due to heat stress and off-gassing .....	4
Table 2. Off-gassing of phenol, naphthalene, aniline, and quinoline from heat stressed ACM .....	4
Table A-1. Summary of ACM Analysis .....	9
Table A-2. Tentative identification and quantification of semivolatile compounds released from heat stressed ACM .....	10

## PREFACE

The research reported herein was initiated and conducted at the Operational Toxicology Branch of the Air Force Research Laboratory under Air Force contract number F41624-96-C-9010 at Wright-Patterson Air Force Base.

Special acknowledgments and appreciations are extended to the following individuals (listed in alphabetical order): Dr. Frank Abernathy, CPT Jody Cline, SPC Jennifer Decatur, Dr. Linda Graeter, Maj Lana Harvey, SGT Ernie Hiltz, Dr. Edgar Kimmel, SGT Donald Kotulan, Mr. Karl Kuhlmann, Lt Col Barbara Larcom, Mr. Willie Malcomb, Mr. Dean Norton, SPC Robert Peterson, Mr. Jim Reboulet, Mr. William Sonntag, and Dr. Kirk Yerkes.

There were three distinct project phases. The preliminary work (Phase 0) addressed the physical behavior of burning advanced composites, such as mass loss rates and smoke plume dispersion. Results of Phase 0 were presented in AFIT/GEEM/ENV/94S-21 (Roop, 1994).

The second phase looked at the chemical and morphological properties of the smoke produced by burning advanced composite material in a small-scale wind tunnel. Results from these experiments were presented in AL/OE-TR-1996-0124.

This technical report presents the off-gassing findings from the final phase. This work analyzed the products released following the heat stress of advanced composite materials used on high performance aircraft.

## ABBREVIATIONS

ACM	Advanced Composite Materials
BMI/CF	Bismaleimide Carbon Fiber
°C	Degrees Celsius
CASRN	Chemical Abstracts Service Registry Number
d#	Deuterium atom on the “#” carbon ( <i>e.g.</i> , aniline-d <sub>5</sub> means the deuterium is attached to the fifth aniline carbon)
EPA	US Environmental Protection Agency
g	Gram
GC/MS	Gas chromatography/mass spectroscopy
L/min	Liters per minute
min	Minute
mL	Milliliter
ug	Micrograms



## INTRODUCTION

Several military systems, especially aircraft, contain one or more types of advanced composite material (ACM) in major and/or minor structural components. Additionally, both European and American-made commercial aircraft are now using ACM in their construction. For example, the Airbus A310 contains approximately 1,100 pounds of ACM and the Boeing 777 contains approximately 33,000 pounds (Stover, 1994). These advanced composite materials allow distinct advantages over metal components in such characteristics as electromagnetic transparency, minimal thermal expansion, corrosion resistance, and reductions in structural stress and fatigue. While these materials offer unique engineering benefits over previously used materials, their chemical and physical nature may predispose them to release toxic compounds when thermally stressed as might occur during a mishap.

Comprehensive environmental, safety, and occupational health constraints dictate that potential toxic effects of new materials and the possible hazards associated with employed manufacturing and maintenance technologies be considered during the design process. Addressing these issues during the system design minimizes the need for post-production retrofits to ensure operator safety and health.

The release of potentially toxic compounds during the combustion of an ACM containing bismaleimide carbon fibers (BMI/CF) has been demonstrated (Courson et al., 1996; Lipscomb et al., 1997). Potential off-gassing from thermally stressed ACM is a concern since follow-on activities after a mishap include collection, evaluation, and possible repair of damaged materials. In addition, the collected material is frequently stored in an enclosed space (*e.g.*, a hangar). The objective of the work presented in this report was to determine if a thermally stressed ACM continues to off-gas and, if so, to characterize the released components.

## MATERIALS AND METHODS

**ACM Samples:** The ACM samples were received from the manufacturer, Lockheed-Martin Aeronautical Systems Company (Marietta, GA) through the F-22 Systems Program Office at Wright-Patterson Air Force Base, Ohio. Several types of ACM were received for analysis. Four samples (manufacturer's identification: 5HF3954, JR-97-313, JR-97-310, and JR-94-029) were selected for analysis because the quantity of material received was sufficient to allow extensive testing. Each sample was assigned a single-letter identifier [5HF3954 (Sample A), JR-97-313 (Sample B), JR-97-310 (Sample C), and JR-94-029 (Sample D)]. Samples A, B, and C appeared to be BMI/CF types of ACM (no composition information was provided for any of the samples). Sample D appeared to be a metal composite, rather than a BMI/CF based composite.

**Thermolyne muffle furnace:** A Thermolyne muffle furnace (Dubuque, IA) was used to heat the ACM samples to 625°C. The heating chamber of the furnace did not have a supply of O<sub>2</sub> to support combustion. The furnace was placed under an exhaust hood to prevent airborne contamination of the laboratory.

**Stainless steel canisters:** A separate stainless steel canister (Figure A-1) was made for each sample to be tested. These canisters (0.9 -1.1 liters each) were equipped with a stainless steel nipple on each end for the purging inlet and outlet. The nipples were fitted with threaded caps to seal the canisters between purges. All canisters were tested to ensure their ability to maintain pressure. Canister #3 failed and was not used.

**Thermal Stress:** The mass of each ACM sample was measured and individual masses ranged from 10g to 50g (Table A-1). Samples A, C, and D were placed individually into the furnace and heated to 625°C. Each sample was held at this temperature until smoke was detected escaping from the oven (30-60 seconds). Sample heating was terminated by opening the furnace door. The sample was removed from the furnace and placed in a canister. The canister was sealed and maintained at room temperature (19°C to 25°C). Post-burn masses of the samples were measured after the last purge cycle (Table A-1).

The same procedure was attempted with sample B but the material liquefied in the furnace. Researchers were unable to transfer the entire sample from the furnace to the stainless steel canister (canister 8). Aluminum boats were fabricated to contain the remaining two B samples (canisters 9 and 10). Even though aluminum's melting point of 661°C is close to the furnace operating temperature, it was selected to minimize the tare weight.

**Purging Methods:** Canisters were purged once a day for 15 or 16 days. Immediately prior to purging, the cap was removed from the inlet nipple and the purge line (refrigeration-grade copper tubing) was quickly connected. The outlet nipple was uncapped and quickly connected to either a Tenax TA<sup>®</sup> tube (7 samples) or a cold trap (7 samples). Helium was delivered at 20-35 mL/minute for 100 minutes. This flow rate and duration produced a purge volume sufficient to replace the canister volume approximately twice during each purge cycle. Individual rate of flow to each canister was controlled through the use of a micro-metering valve and flow rate was monitored twice per purge. Following the purge, the inlet and outlet nipples were disconnected and capped, the purge line was plugged, and the Tenax TA<sup>®</sup> tube or the cold trap was plugged.

**Tenax TA<sup>®</sup> tube sample analysis:** Tenax TA<sup>®</sup> tube samples were collected in stainless steel tubes containing Tenax TA<sup>®</sup> (Supelco, Bellefonte, PA), a medium designed to trap organic vapors for

chemical analysis. Samples were collected in the Tenax TA<sup>®</sup> tubes as described above. Prior to analysis the tubes were spiked with a mixture containing an internal standard (1,4-dichlorobenzene-d<sub>4</sub>), and surrogate compounds (4-bromofluorobenzene, aniline-d<sub>5</sub>, phenol-d<sub>5</sub>, and nitrobenzene-d<sub>5</sub>) (ChemService, Westchester, PA). The surrogates and internal standard were dissolved in purge-trap grade methanol (Fisher Scientific, Fair Lawn, NJ). [Note: d<sub>#</sub> in chemical nomenclature indicates the replacement of a hydrogen atom on the “#” carbon of the parent compound with a deuterium atom.]

A surrogate is a compound added to the system after sample collection and is used to determine extraction efficiency for the compound of interest. Para-bromofluorobenzene (4-bromofluorobenzene) is an EPA surrogate for the volatile fraction. Aniline-d<sub>5</sub> and phenol-d<sub>5</sub> were selected as deuterated analogs for quantitation. Nitrobenzene-d<sub>5</sub> was included as an alternate internal standard in case of interference with primary internal standard (e.g. coelution of two compounds). The Tenax TA<sup>®</sup> were loaded onto a Perkin-Elmer ATD 400 autosampler/thermal desorption unit (Perkin Elmer Corporation, Norwalk, CT) connected to Perkin-Elmer Q-Mass 910 gas chromatograph/mass spectrometer. Compound separation was accomplished on a DB-5 (J & W Scientific, Folsom, CA) capillary column.

Four compounds (phenol, aniline, quinoline and naphthalene) were selected for detailed analysis because they were identified in previous work with a BMI/CF type of ACM and because they are the most structurally-simple members of four individual series of homologous compounds. For example, aniline is the simplest member of the series of aromatic amines, phenol is representative of the aromatic alcohols, naphthalene is representative of the polycyclic aromatic hydrocarbons and quinoline represents the nitrogen-containing polycyclic aromatic hydrocarbon equivalents. Their selection does not imply that these chemicals are those most commonly encountered, most toxic, or most readily detectable.

Calibration curves were established for these four compounds and the resulting relative response factors were used to quantify each compound in the sample per EPA Method 8260 (EPA, 1986). Ion match utilizing the Wiley 5th edition library (Palisade Mass Spectrometry, Newfield, NY) tentatively identified other peaks in the chromatogram. These tentatively identified compounds were quantified by applying a uniform response factor of 1.0 as compared to the total response of the internal standard.

Cold trap sample analysis: Prior to purging, the first U-tube (Figure A-2) was placed in a pocket heater (O.I. Corp, College Station, TX) at 80°C and the second U-tube was pre-cooled by immersion in liquid nitrogen for ten minutes prior to flow initiation. This U-tube was maintained under liquid nitrogen for the duration of the purging cycle. The cold trap was flushed with helium (via a stainless steel transport line) for 30 minutes at approximately 10 mL/min.

During canister purging, the outlet of the low-volume trap passed through a small amount of methylene chloride to capture materials that might escape the cold trap. After purging, the tube was flushed with several small amounts of methylene chloride, and the final sample (the methylene chloride of the final trap plus the flush) made up volumetrically to 1 mL. Internal standards (1,4-dichlorobenzene-d<sub>4</sub>, naphthalene-d<sub>8</sub>, acenaphthene-d<sub>10</sub>, phenanthrene-d<sub>10</sub>, chrysene-d<sub>12</sub>, and perylene-d<sub>12</sub>) were added to the collected methylene chloride. Analysis of the methylene chloride was performed on a Perkin-Elmer Q-mass 910 GCMS system.

## RESULTS

ACM samples were thermally stressed by heating to 625°C at ambient pressure. Heating time was the approximate time between closing the furnace door and smoke emission. All samples autoignited after the oven door was opened exposing the sample to normal atmospheric oxygen. Flames were extinguished by transferring the burning sample to a stainless steel canister and sealing the canister with the lid. Heating of ACM resulted in the following loss of mass (Table 1).

Sample	Reduction in Mass (Avg)	Approximate Heating Time (Seconds)
A	16%	30
B	3.2%	45
C	10%	30
D	2.1%	60

**Table 1. Average reduction of sample ACM mass due to heat stress and off-gassing.**

Samples collected from the off-gassing of the thermally stressed ACM were analyzed for phenol, naphthalene, aniline, and quinoline (Courson, *et al.*, 1996). The average amount of these semivolatile compounds off-gassed per day for the first five days is presented in Table 2.

Component	Samples Shown in ng/gram Off-Gassed Per Day			
	Sample A (n=4)	Sample B (n=4)	Sample C (n=4)	Sample D (n=2)
Phenol	7.48 ± 1.38 (0.987)	0.80 ± 0.11 (0.998)	519 ± 330 (0.969)	35.1 ± 13.1 (0.905)
Naphthalene	nd	0.16 ± 0.07 (0.949) <sup>1</sup>	81.8 ± 28.0 (0.995)	895 ± 141 (0.986)
Aniline	nd	0.26 ± 0.12 (0.998)	356 ± 236 (0.976)	510 ± 77 (0.979)
Quinoline	nd	nd	8.6 ± 4.8 (0.926)	29.4 ± 5.3 (0.963)

**TABLE 2. Off-gassing of phenol, naphthalene, aniline, and quinoline from heat stressed ACM.**

Relative rates of off-gassing of four representative chemicals. Data are presented as mean ± standard deviation, ng component off-gassed per gram encased material per day, calculated over the initial 5 days. Regression coefficients are presented in parenthesis. nd = compound not detected.

<sup>1</sup> The amount released demonstrated an appreciable reduction at approximately 4 days.

The semivolatile compounds tentatively identified by ion match are presented in Table A-2. The quantification is an estimate because a uniform response factor of 1.0 was used to compare each compounds response to the internal standard.

In both analyses (specific and ion match), off-gassed compounds were detected in the final purges of the canisters for each sample of ACM, albeit at much lower levels than initial canisters.

## SUMMARY

This preliminary study looked at the semivolatile compounds off-gassed from four different types of ACM following thermal stress. Finding off-gassed compounds in the final purges of the canisters indicates that, under similar conditions, these four types of ACM will off-gas for at least two weeks following thermal stress at 625°C. Results with the four targeted compounds (aniline, phenol, quinoline, and naphthalene) identified both qualitative and quantitative differences (Table 2.) in the off-gassing of potentially toxic components. These qualitative and quantitative differences were further supported by the data on the semivolatile compounds tentatively identified by ion match (Table A-2). These qualitative and quantitative differences in the off-gassing from the four different types of ACM test materials indicate that it would be imprudent to extrapolate the off-gassing characteristics of a specific compound to another type of ACM. However, it can be deduced that only well-ventilated areas should be used for thermally stressed ACM to prevent the build-up of off-gassed materials to dangerous levels. In addition, the study results fully support the continued use of the personal protective equipment guidelines presented in paragraph 3-4,e,(3) of TO 00-105E-9, Aircraft Emergency Response.

These results underscore the need to examine the specific ACM in question and address the issues associated with that ACM. Further research is recommended to fully characterize ACM off-gassing following thermal stress.

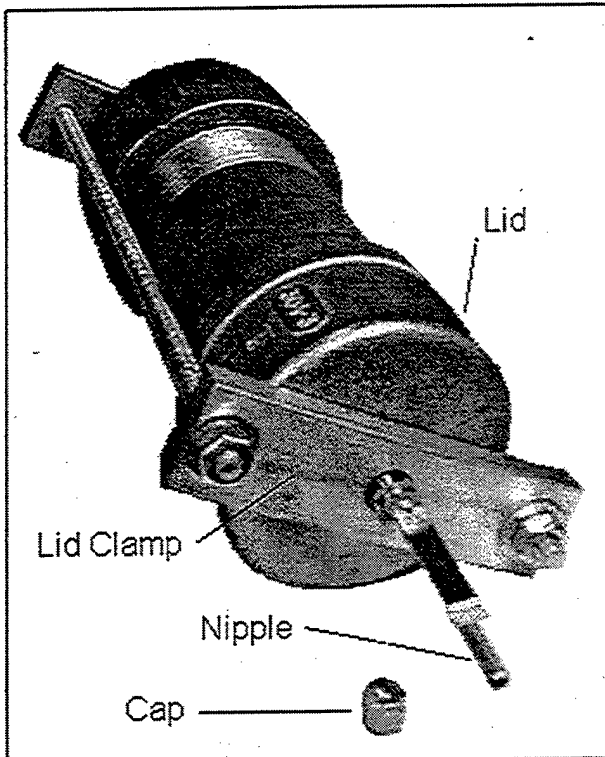
## REFERENCES

Courson, Flemming, C.D., Kuhlmann, K.J., Lane, J.W., Grabau, J.H., Cline, J.M., Miller, C.R., Larcom, B.L., and Lipscomb, J.C. (1996). Smoke production and thermal decomposition products from advanced composite materials WRAIR-TR-1995-0027; AL/OE/TR-1996-0124.

Lipscomb J.C., Kuhlmann, K.J., Cline, J.M., Larcom, B.J., Peterson, R.D., Courson, D.L. Combustion Products From Advanced Composite Materials. *Drug Chem Toxicol*, 20(4), 281-292, (1997).

Stover, D. Composites in Large Commercial Planes. *High Performance Composites* July/Aug: 18-21, 1994.

Test Methods for Evaluating Solid Waste: Physical/Chemical Methods Nov 1986; United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington D.C., 20460.



**Figure A-1. Canister.**

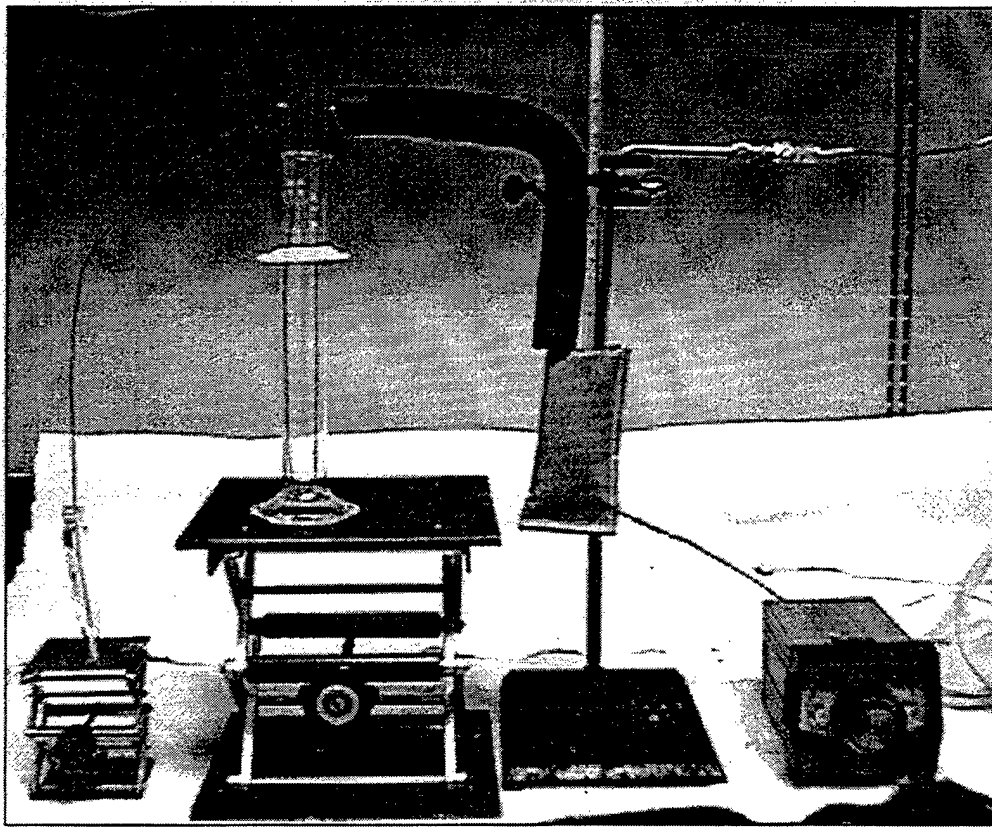


Figure A-2. Cold Trap Set-Up.



Canister	Sample	Mfr ID <sup>1</sup>	Time at (625°C)	Collection Method	Mass (initial)	Mass (end)	Mass Lost	Number purges (1 per day)
1	C	JR-97-310	30 sec	Cold Trap	34.23 g	32.13 g	2.10 g	15
2	C	JR-97-310	30 sec	Cold Trap	36.64 g	34.96 g	1.68 g	15
3	Not Used – canister failed operational criteria test							
4	A	5HF3954	30 sec	Cold Trap	10.89 g	8.85 g	2.04 g	15
5	D	JR-94-029	60 sec	Tenax	45.94 g	45.00 g	0.94 g	15
6	C	JR-97-310	30 sec	Tenax	33.96 g	29.52 g	4.44 g	16 <sup>2</sup>
7	D	JR-94-029	60 sec	Cold Trap	42.72 g	41.38 g	1.34 g	15
8	B	JR-97-313	45 sec	Tenax	50.03 g	NA <sup>3</sup>	NA <sup>3</sup>	16 <sup>2</sup>
9	B	JR-97-313	45 sec	Tenax	51.91 g	50.14 g	1.77 g	15
10	B	JR-97-313	45 sec	Cold Trap	47.32 g	45.16 g	2.16 g	15
11	A	5HF3954	30 sec	Cold Trap	11.69 g	9.89 g	1.80 g	15
12	B	JR-97-313	45 sec	Cold Trap	45.94 g	45.29 g	0.65 g	15
13	C	JR-97-310	30 sec	Tenax	35.36 g	32.85 g	2.51 g	15
14	A	5HF3954	30 sec	Tenax	12.51 g	10.55 g	1.96 g	15
15	A	5HF3954	30 sec	Tenax	10.50 g	8.78 g	1.72 g	15

**TABLE A-1. Summary of ACM Analysis.** The physical appearances of samples A, B, and C were consistent with being ACM. Sample D appeared to be a metal. Sample thickness varied by the number and type of coatings. Each sample used was cut to make a coupon *approximately* 38.1 mm wide X 76.2 mm long X 1.8 mm thickness.

<sup>1</sup> Manufacturer's identification number for the sample.

<sup>2</sup> The samples in canisters #6 and #8 were purged an additional day because they were used in setting GC/MS and Thermal Desorption unit split ratios.

<sup>3</sup> Sample B in canister 8 left residue on the floor of muffle furnace that couldn't be recovered.

CASRN	NAME	ng/5Days				ng/5days/gram			
		A	B	C	D	A	B	C	D
000000-00-0	Methyl Ester of 2-Oxo Hexanoic acid		25532				493		
000000-00-0	2,6 8-Trimethylnon-4-on-5-ene		16031	3908			320	111	
000000-00-0	Piperinocyclohexene		12842				248		
000000-00-0	Methyl-E-(2,2,3,3-Tetramethylcyclopropyl		8104	12528			156	354	
000000-00-0	2,6,8-Trimethyl-4-nonanone			6364				180	
000000-00-0	N-Octan-3-ene			99				3	
000000-00-0	2,4-Dimethylcyclopent-4-ene-1,3-dione			21				1	
000071-41-0	1-Pentanol				55				1
000071-43-2	Benzene	55	51513	544747	72375	5	1030	16041	1575
					6024				131
000078-83-1	1-Propanol, 2-methyl-		5009	32147	8040		100	909	175
000078-93-3	2-Butanone (MEK)		10576	7564	17368		211	223	378
000087-59-2	Benzenamine, 2,3-dimethyl-	77				7			
000090-12-0	Naphthalene, 1-methyl-			24				1	
000095-01-2	Benzaldehyde, 2,4-dihydroxy-			76				2	
000095-13-6	1H-Indene		20647	6923	10324		413	196	225
000095-47-6	Benzene, 1,2-dimethyl-O-xylene		2.5E+07	267582			488777	7567	
			75				1		
000095-48-7	Phenol, 2-methyl-			6075				179	
000095-87-4	Phenol, 2,5-dimethyl-	2346				223			
000095-93-2	Benzene, 1,2,4,5-tetramethyl-		36				1		
000098-06-6	Benzene, (1,1-dimethylethyl)-		38				1		
000098-82-8	Benzene, (1-methylethyl)-		86158	150023			1664	424	
			17946				359		
000100-41-4	Benzene, ethyl-	79	304561	125430	8647	8	5882	3547	188
000100-42-5	Styrene			8067	23560			238	513
000100-47-0	Benzonitrile		5450				109		
000100-61-8	Benzenamine, N-methyl-		7567				151		
000103-65-1	Benzene, propyl-		9555				184		

**Table A-2. Tentative Identification and Quantification (Average) of Semivolatile Compounds Released From Heat Stressed ACM.**

CASRN	NAME	ng/5Days				ng/5days/gram			
		A	B	C	D	A	B	C	D
000103-69-5	Benzenamine, N-ethyl-				48				1
000104-76-7	1-Hexanol, 2-ethyl-			11662	23432			330	510
000106-35-4	3-Heptanone		6722				134		
000106-42-3	Benzene, 1,4-dimethyl-P-xylene		5221367		20444		100838		445
			2149081				42956		
000107-00-6	1-Butyne			3582				105	
000107-87-9	2-Pentanone		11380				228		
000108-10-1	2-Pentanone, 4-methyl-	3028	>1000000	>21487419	38929	288 *	*		847
			>9527430				*		
000108-11-2	2-Pentanol, 4-methyl-			14030				397	
000108-38-3	Benzene, 1,3-dimethyl-MO-xylene		864831	510750	29033		17286	14444	632
				8006				236	
000108-83-8	4-Heptanone, 2,6-dimethyl-			82				2	
000108-88-3	Benzene, methyl-(Toluene)		65263	47660	42886		1304	1403	934
			7522				145		
000108-90-7	Benzene, chloro-			20893				591	
000108-96-3	4(1H)-Pyridinone		44				1		
000109-06-8	Pyridine, 2-methyl-			97				3	
000110-65-6	2-Butyne-1,4-diol	15				1			
000110-86-1	Pyridine				62729				1365
					31522				686
000115-11-7	1-Propene, 2-methyl-			48451				1370	
000120-92-3	Cyclopentanone			3827				108	
000121-69-7	Benzenamine, N,N-dimethyl-		6637				133		
000123-91-1	1,4-Dioxane		11132	27898	60613		223	789	1319
000142-82-5	Heptane			5162				152	
000151-10-0	Benzene, 1,3-dimethoxy-		84				2		
000271-89-6	Benzofuran			39257				1156	
000286-62-4	9-Oxabicyclo[6.1.0]nonane				71				2
000292-64-8	Cyclooctane			4450	8664			126	189

**Table A-2, cont'd. Tentative Identification and Quantification (Average) of Semivolatile Compounds Released From Heat Stressed ACM.**

CASRN	NAME	ng/5Days				ng/5days/gram			
		A	B	C	D	A	B	C	D
000300-57-2	Benzene, 2-propenyl-			65				2	
000470-99-5	2-Cyclohexen-1-ol, 3,5,5-trimethyl-		20150				389		
000497-26-7	1,3-Dioxolane, 2-methyl-				8680				189
000502-44-3	2-Oxepanone		40566	118416			811	3349	
			6501	12716			126	374	
000502-56-7	5-Nonanone		11630				225		
000503-64-0	2-Butenoic acid, (Z)-				4850				106
000513-42-8	2-Propen-1-ol, 2-methyl-			13516	16849			382	367
000526-73-8	Benzene, 1,2,3-trimethyl-		8509				164		
000526-75-0	Phenol, 2,3-dimethyl-	2119				202			
000527-60-6	Phenol, 2,4,6-trimethyl-	3867				309			
000536-74-3	Benzene, ethynyl-		13150	5835	8158		263	165	178
000576-26-1	Phenol, 2,6-dimethyl-	7065				565			
000589-92-4	Cyclohexanone, 4-methyl-				6049				132
000591-47-9	Cyclohexene, 4-methyl-			8645				255	
000591-76-4	Hexane, 2-methyl-			5484				155	
000591-78-6	2-Hexanone			6331				186	
000592-43-8	2-Hexene		32348	62172			647	1831	
000598-61-8	Cyclobutane, methyl-			10137				287	
000611-14-3	Benzene, 1-ethyl-2-methyl-		21729				420		
000620-14-4	Benzene, 1-ethyl-3-methyl-		15737		55		304		1
000622-96-8	Benzene, 1-ethyl-4-methyl-		5946		46		115		1
000623-56-3	3-Hexanone, 5-methyl-		42744	33016			854	934	
000628-73-9	Hexanenitrile		7004	21580	11075		140	635	241
				5189				147	
000697-82-5	Phenol, 2,3,5-trimethyl	32				3			
000764-48-7	Ethanol, 2-(ethenyloxy)-			4407	6150			125	134
000814-78-8	3-Buten-2-one, 3-methyl-				5148				112
000823-22-3	2H-Pyran-2-one, tetrahydro-6-methyl-	62				6			

**Table A-2, cont'd. Tentative Identification and Quantification (Average) of Semivolatile Compounds Released From Heat Stressed ACM.**

CASRN	NAME	ng/5Days				ng/5days/gram			
		A	B	C	D	A	B	C	D
000868-77-9	2-Propenoic acid, 2-methyl-, 2-hydroxyethyl ester			19720				558	
000930-56-3	Ethanone, 1-(2-methylcyclopropyl)-		80				2		
000934-80-5	Benzene, 4-ethyl-1,2-dimethyl-		38				1		
001072-05-5	Heptane, 2,6-dimethyl-		7166				138		
001120-72-5	Cyclopentanone, 2-methyl-		9472	6099	8863		189	180	193
001121-84-2	2H-Pyran-2-one, tetrahydro-4-methyl-	9044	205191	272104	521313	723	4101	7695	11348
			8554	187856	302691		165	5313	6589
001330-20-7	Xylene		836708	103734			16724	2934	
			82236				1644		
001632-16-2	Heptane, 3-methylene-				21376				465
001679-49-8	2(3H)-Furanone, dihydro-4-methyl-		71134				1422		
001927-69-1	2H-Pyran, 2-(1,1-dimethylethoxy) tetrahydro-		18534				358		
002198-23-4	4-Nonene		78				2		
002244-07-7	Undecanenitrile				89				2
002849-98-1	2-Propenoic acid, 2-methyl-, pentyl ester		26969				539		
003160-38-1	4-Methylbenzalacetone			6610				195	
003749-51-7	2(1H)-Pyridinone, 4-hydroxy-6-methyl-		50837				982		
003973-18-0	Ethanol, 2-(2-propynyloxy)-				4705				102
004050-45-7	2-Hexene, (E)-			20274				597	
				4776				135	
004265-25-2	Benzofuran, 2-methyl-			12342				363	
004316-48-7	Methyl 8-oxooctanoate			48401				1369	
004655-34-9	Methacrylic acid, isopropyl ester		78368	87771	203439		1566	2482	4428
005204-80-8	4-Pentenal, 2-ethyl-			30886				873	
005363-63-3	Pentane, 1-(ethenyloxy)-				45				1
005685-46-1	1,1'-Bicyclopropyl			3474				102	
005754-18-7	3,6-Pyridazinedione, 1,2-dihydro-4-methy			42				1	
007045-71-8	Undecane, 2-methyl-		97				2		
007642-04-8	2-Octene, (Z)-				30039				654

**Table A-2, cont'd. Tentative Identification and Quantification (Average) of Semivolatile Compounds Released From Heat Stressed ACM.**

CASRN	NAME	ng/5Days				ng/5days/gram			
		A	B	C	D	A	B	C	D
010240-08-1	1-Naphthalenol, 4-methyl-	26				3			
010374-14-8	Cyclobutanone, 2-ethyl-				36				1
013228-36-9	1H-Indole, 5-methyl-2-phenyl-	72				7			
013269-52-8	3-Hexene, (E)-			5057				149	
014919-01-8	3-Octene, (E)-			70				2	
017059-52-8	Benzofuran, 7-methyl-			6762				199	
017257-83-9	4-Octanone, 2,3-epoxy-2-methyl-				47				1
017302-28-2	Nonane, 2,6-dimethyl-		70				1		
018402-82-9	3-Octen-2-one, (E)-				58				1
018733-57-8	Silane, trichloroecicosyl-		93				2		
026952-21-6	Isooctanol			71				2	
028290-01-9	Cyclobutanone, 2,3,3-trimethyl-				12329				268
029350-67-2	Cyclohexene, 1-methyl-4-(1-methylethyl)-		49				1		
036917-36-9	Pyridine, 4-ethyl-2,6-dimethyl-	26				2			
040499-83-0	3-Pyrrolidinol			9838				278	
049607-51-4	1H-1,2,4-Triazol-3-amine, 1-methyl-	89				8			
053864-08-7	1,3-Butadienyldiene) cyclohexane		31				1		
055000-49-2	Benzoic acid, 2,5-dimethyl-, (2,5-dimethylphenyl) methyl ester				39				1
055444-09-2	5-Acetoxy-3,4,4-Trimethyl-2-Cyclopenten-1-one		9323				180		
056745-74-5	2-Acetyl-3-N-Butylcyclopentanone		22067				426		
059920-26-2	2,4,6,8-Tetramethyl-1-undecene				56				1
061142-13-0	1-Propene, 1-(2-propenyloxy)-, (E)			40				1	
062338-57-2	1,4-Cyclohexadiene, 3-ethenyl-1,2-dimeth		23				0		
072101-22-5	2-Propenal, (1,1-dimethylethyl)methyl				96				2
077572-68-0	Butanal, 3,3-dimethyl-2-oxo-, hemihydrate		6552				131		
077822-51-6	(Z)-3-Isopropyl-3,6-dimethyl-4,6-heptadien-2-one		88				2		
			84				2		
080839-92-5	(E)-1-(1-Butenyl)aziridine				93				2

**Table A-2, cont'd. Tentative Identification and Quantification (Average) of Semivolatile Compounds Released From Heat Stressed ACM.**